The first x-ray diffraction peak of water as a function of temperature in the range 4 – 63 °C

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X-ray and neutron diffraction are commonly used to investigate the structure of water under varying physical conditions [1-3]. Here we report a preliminary experiment to measure the position and amplitude of the first x-ray diffraction peak (Q_0) of water as a function of temperature. The experiment was performed on the SAXS/WAXS beamline of the Australian Synchrotron with a Pilatus 1M SAXS (1,000 × 1,000 pixel) camera. The wavelength of the x-rays was 0.61992 ± 0.00003 Å, corresponding to an energy of 20.026 ± 0.005 keV. Distilled/deionised water flowed between a tempering beaker immersed in a temperature controlled water bath and a 1.5 ± 0.1 mm quartz capillary placed in the x-ray beam (figure 1). Thermocouples were placed in the tubing either side of the capillary to estimate the temperature at the point where the beam passed through the capillary. A sequence of 10 s integrations was obtained as the temperature of the water was raised from 4 – 63 °C. Figure 2 shows the structure function for the air-filled and water-filled capillary at 25 °C and the curve produced by a subtraction of the former from the latter. We compared our data with structure functions obtained on the Advanced Light Source (ALS) using a 11 keV beam glancing off a 0.5 mm thick sample of water in a temperature controlled chamber [3]. Figure 3 shows a plot of the first structural peak (Q_0) as a function of temperature for the AS and ALS, and figure 4 shows a plot of the normalised intensity of Q_0 as a function of temperature for the ALS and AS. Both sets of data show that the position of the peak increases with temperature whilst the amplitude decreases. Further experiments are required to ascertain if the subtle differences seen between the AS and ALS structure functions are instrumental or due to differences between the structure of water confined to a 0.5 mm thick flat sheet and flowing in a 1.5 mm diameter capillary.

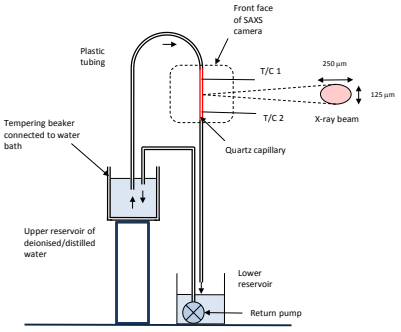

Figure 1. Schematic diagram of the apparatus. The FWHM dimensions of the x-ray beam are given.

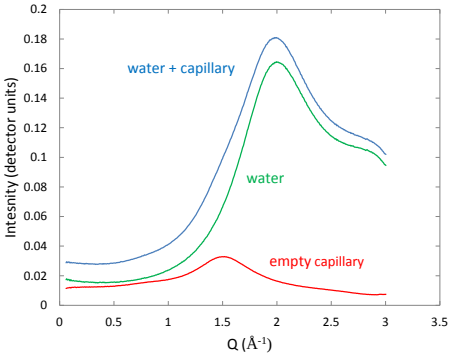

Figure 2. Structure functions of the air-filled capillary, water-filled capillary and subtraction of the two, at 25 °C.

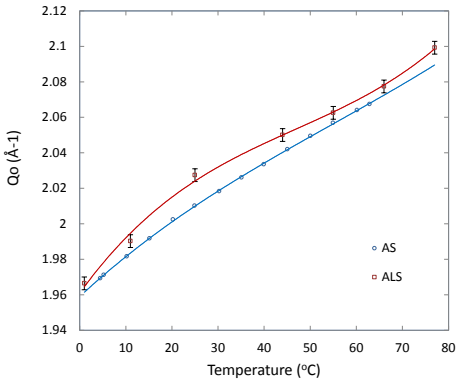

Figure 3. Q_0 as a function of temperature for data obtained using the Advanced Light Source (ALS □) and the Australian Synchrotron (AS ○). A third order polynomial has been fitted to both sets of data to guide the eye. The ALS error bars are ± 0.0036 Å^{-1}. The majority of the AS error bars are ± 0.0002 Å^{-1} with some at ± 0.0003 Å^{-1}. These have not been plotted as they are only about one third the vertical height of the circle symbols and therefore too small to be seen.

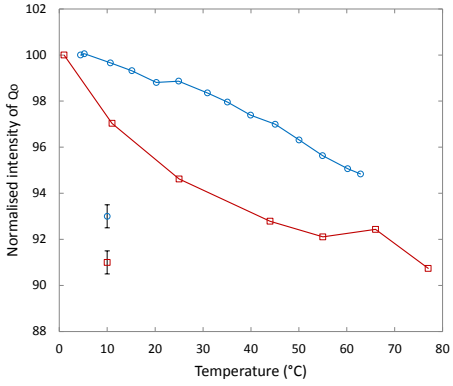

Figure 4. Plot of the normalised intensity of Q_0 as a function of temperature for the ALS and AS. In both cases the error bars are about 0.5% of the amplitude. For both sets of data the intensity of the structure function at the lowest temperature (1 °C for the ALS and 4 °C for the AS) was taken as 100%.

References. 1. Narten, A.H., Danford, M.D., Levy, H.A. X-ray diffraction study of liquid water in the temperature range 4-200 °C. *Discuss. Faraday Soc.*,**43**, 7-107 (1967) DOI: 10.1039/DF9674300097 2. Soper, A.K. The radial distribution functions of water and ice from 220 to 673 K and at pressures up to 400 MPa. *Chem. Phys.* **258**, 121-137 (2000). 3. Hura, G. Sorenson, J. M., Glaeser, R.M., Head-Gordon, T. A high-quality x-ray scattering experiment on liquid water at ambient conditions. *J. Chem. Phys.* **113**, 9140-9148 (2000). **Acknowledgments.** The Australian Synchrotron is thanked for funding this work.